



RESEARCH ARTICLE

# Catalytic Downflow Liquid Contact Reactor for Biomass Pretreatment To Biofuel Production

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## ABSTRACT

The quest for alternative energy generation sources that are inexpensive, eco-friendly, renewable and can replace fossil fuels is due to the increasing energy demands. Production of biofuels from lignocellulosic biomass holds the remarkable potential to meet the current energy demand as well as to mitigate greenhouse gas emissions for a sustainable clean environment. Lignocellulose biomass feedstocks compulsorily undergo pretreatment process to release the fermentable sugars. Most of the existing pretreatment processes are energy-intensive and consume more cost. Low cost and effective pretreatment are essential to overcome the hurdles in commercializing biofuel production from lignocellulose biomass. Catalytic Downflow liquid contact reactor (CDLCR) is an innovative method used to pretreat the lignocellulosic biomass, consisting of a pump, electrical motor, circulation tank and pipe accessories. The present study deals with pearl millet biomass pretreatment using alkalicatalyst ( $\text{Ca}(\text{OH})_2$ ) in CDLCR under ambient conditions. The experimental trial results show that there was a significant reduction in lignin and hemicellulose observed for pearl millet biomass. SEM and FTIR analysis supports and confirms these results. The highest lignin reduction were achieved as 69 % via optimal conditions (lime: 12 %, reactor column height: 1.2m and reaction period: 60min.). Enzymatic saccharification studies show the highest reducing sugar yield was  $40.6 \pm 0.6 \text{ mg g}^{-1}$  in 96 h at 40 FPU/g. This innovative pretreatment reactor was more effective for lignin removal.

**Keywords:** Lignocellulosic biomass; Pretreatment; Catalytic Downflow Liquid Contact Reactor

## INTRODUCTION

Lignocellulosic biomass feedstocks are mainly made of a mixture of carbohydrate polymers viz., cellulose, hemicelluloses, and lignin. Production of biofuels, especially bio-ethanol from lignocellulosic biomass, holds the remarkable potential to meet the current energy demand. Pearl millet (*Pennisetum glaucum*) is a warm-season annual grass and grows in semiarid conditions with very low ( $\leq 300\text{mm}$ ) or inconsistent rainfall. Pearl millet serves as a good offset for biofuel production. The basic process steps in producing bioethanol from lignocellulosic materials are pretreatment, hydrolysis, fermentation and product separation/distillation. So, pretreatment is one of the main preprocessing operations for lignocellulosic biomass-based biofuel production. Many pretreatment technologies based on numerous physical, chemical, and biological methods have been developed, altering/damaging lignocelluloses' structure and

removing lignin (Chaturvedi and Verma, 2013). In the alkaline treatment, biomass is treated with an alkali such as sodium, potassium, calcium and ammonium hydroxides at normal temperature and pressures.

The main advantage of the process is the efficient removal of lignin from the biomass. The cost of lime required to pretreat a given quantity of biomass is the lowest among alkaline treatments (Chang and Holtzapple, 2000). The study aims to optimize conditions for lignin reduction of pearl millet biomass in catalytic downflow liquid contact reactor and test results are presented in this paper.

## MATERIAL AND METHODS

The pearl millet biomass variety CO7 was collected, dried at the ambient temperature and powdered with a shredder and grinder's help. The size of the biomass particles used in this experiment was  $212 \mu\text{m}$ . A catalytic Downflow liquid contact reactor was developed and used in this study. In this

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reactor, there is no mechanical device used mixing. It consists of a pump, electrical motor, circulation tank and pipe accessories. The biomass slurry was prepared by mixing of catalyst, powdered biomass sample and water. In order to make a closed-loop circulation the biomass slurry was supplied from the circulation tank to there actor column with the help of a centrifugal pump and returned to the circulation tank. The electrical power required for an operation of this reactor was found as 373W. The conditions selected and used in the experiment were 8, 10 and 12% of an alkaline catalyst, 10% biomass loading, and reaction time (5, 20 and 60min.). The alkaline catalysts selected for this study were calcium hydroxide and test results are presented. In order

to compare the change in the biomass before and after pretreatment, the FTIR spectra of the raw and pretreated biomass samples were obtained using an FT-IR (FT-IR 6800 JASCO, Japan). Absorbance spectra were recorded between 4000 to 400  $\text{cm}^{-1}$  wave numbers with a spectral resolution of  $4\text{cm}^{-1}$  and 64 scans per sample.

## RESULTS AND DISCUSSION

The Catalytic Down flow liquid contact reactor treatment has a potential effect on biomass pretreatment under milder conditions, providing localized high energy to break down crystalline cellulose and facilitate the formation of radical species to degrade lignin, as shown from the results.

Alkali treatment of lignocellulose disrupts the cell wall by dissolving hemicellulose, lignin, and silica, hydrolyzing uronic and acetic esters, and swelling cellulose. Total reducing sugar (TRS) content in biomass was depicted in the (Table 1).

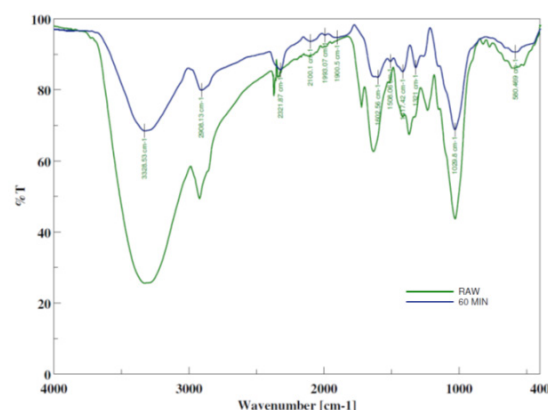
**Table 1- Enzymatic saccharification of raw and pretreated pearl millet biomass**

Lime - 12% loading	Saccharification efficiency				
	24 $\text{hg}^{-1}$	36 $\text{hg}^{-1}$	72 $\text{hg}^{-1}$	96 $\text{hg}^{-1}$	120 $\text{hg}^{-1}$
20 FPU/g	29.45( $\pm$ 0.32) <sup>b</sup>	35.36 ( $\pm$ 0.14) <sup>c</sup>	35.52 ( $\pm$ 0.33) <sup>b</sup>	36.53 ( $\pm$ 0.34) <sup>c</sup>	33.36 ( $\pm$ 0.97) <sup>c</sup>
30 FPU/g	33.45( $\pm$ 0.21) <sup>a</sup>	36.47 ( $\pm$ 0.27) <sup>b</sup>	37.33 ( $\pm$ 0.31) <sup>b</sup>	38.55 ( $\pm$ 0.97) <sup>b</sup>	36.45 ( $\pm$ 0.11) <sup>b</sup>
40 FPU/g	35.43( $\pm$ 0.37) <sup>a</sup>	38.45 ( $\pm$ 0.39) <sup>a</sup>	39.59 ( $\pm$ 0.29) <sup>a</sup>	40.59 ( $\pm$ 0.57) <sup>a</sup>	39.41 ( $\pm$ 1.15) <sup>a</sup>
CV%	1.10	0.60	0.810	0.711	0.53

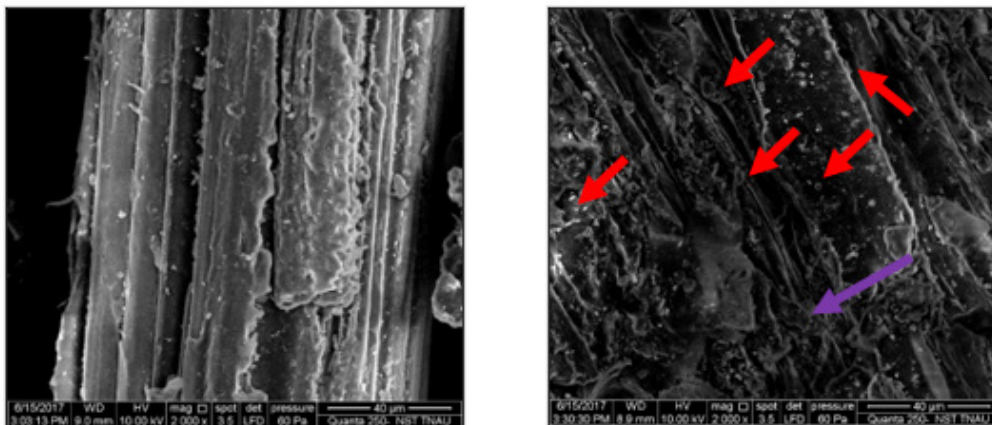
The alkaline pretreatment with 10 percent alkali loading resulted in lignin reduction and part of hemicellulose and increased cellulose reactivity for further hydrolysis steps (Hamelincket *al.*, 2005). The reaction conditions are generally mild, which prevents condensation of lignin, leading to its high solubility and greater removal (Sharma *et al.*, 2012).

Studies showed that in the case of raw biomass samples, clear peaks appeared in the spectra for cellulose, hemicelluloses and lignin with their respective wave numbers, as presented in (Figure1). For alkaline pretreatment in the developed reactor, the rewas evidence for lignin removal. Because the peaks at wave numbers related to lignin compound (1321, 1422, 1509, 1634, and  $3340\text{cm}^{-1}$ ) for the pretreated sample were disappeared or lower peak intensity compared to that of the raw sample. Similarly, there was a low-intensity peak appeared for pretreated samples for hemicellulose compound ( $1157\text{ cm}^{-1}$ ). SEM studies were also performed, which revealed that the raw material has an intact lamellar structure in which cellulose is closely wrapped by hemicellulose and lignin; however, the treated biomass demonstrated ad is tinct inner cavity of cellulose and a disrupted lamellar structure

due to the removal of outer hemicellulose and lignin was shown in (Figure 2).



**Fig. 1. Comparison of FTIR spectra for both raw and pretreated biomass samples**



**Fig. 2. SEM image of raw and pretreated biomass samples**

## CONCLUSIONS

The development of low-cost pretreatment is essential for reducing. Results of the FTIR study showed that there was a reduction in lignin and hemicellulose. The total reducing sugars in the biomass samples were increased with an increase in reaction time. However, additional confirmation studies are required. Further studies on the geometry and construction of the CDLCR system will bring out an efficient high through put system for industrial treatment of lignocellulosic biomass for biofuel production.

## ACKNOWLEDGEMENT

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